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मानक

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“Step Out From the Old to the New”

IS 376 (1986): Sodium hydroxide, analytical reagent [CHD 1: Inorganic Chemicals]



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“Knowledge is such a treasure which cannot be stolen”



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**IS : 376 - 1986**  
**( Reaffirmed 2010 )**

*Indian Standard*  
**SPECIFICATION FOR**  
**SODIUM HYDROXIDE, ANALYTICAL REAGENT**  
*(Third Revision)*

Second Reprint JULY 2007  
(Including Amendment No. 1 & 2)

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**BUREAU OF INDIAN STANDARDS**  
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**AMENDMENT NO. 2 NOVEMBER 2004**  
**TO**  
**IS 376 : 1986 SPECIFICATION FOR**  
**SODIUM HYDROXIDE, ANALYTICAL REAGENT**  
**( Third Revision )**

( Page 7, clause A-4 ) — Substitute the following for the existing clause:

**A-4 DETERMINATION OF CHLORIDES**

**A-4.1 Quality of the Reagents**

Unless specified otherwise, pure chemicals and chloride free deionized or distilled water (*see* IS 1070) shall be used in tests. Use of deionized water is preferable compared to distilled water as it sometimes contains traces of chloride.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

**A-4.2 General**

Two methods have been specified for determination of chlorides by Visual Comparison Method (Method A) and Spectrophotometric Method (Method B). In case of dispute, Method A shall be the referee method.

**A-4.3 Method A (Visual Comparison Method)**

**A-4.3.1 Apparatus**

**A-4.3.1.1 Nessler cylinder** — 50-ml capacity.

**A-4.3.2 Reagent**

**A-4.3.2.1 Dilute nitric acid** — approximately 5 N.

**A-4.3.2.2 Silver nitrate solution** — approximately 4 percent (*m/v*).

**A-4.3.2.3 Standard chloride solution** — Dissolve 0.1649 g of sodium chloride in water and make up the volume to 1 000 ml. Dilute 50 ml of the solution to 100 ml. One millilitre of this solution contains 0.05 mg of chloride (as Cl).

**A-4.3.3 Procedure**

Transfer 10 ml of the sample solution (**A-2.1**) into a Nessler cylinder and dilute with 20 ml of water. Add 6 ml of dilute nitric acid and 1 ml of silver nitrate

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solution. Make up the solution to 50 ml with water. Carry out a control test in the other Nessler cylinder using 1 ml of standard chloride solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the opalescence produced in the two cylinders after 5 minutes.

**A-4.3.4** The limit prescribed for chlorides shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

### **A-4.4 Method B (Spectrophotometry Method)**

#### **A-4.4.1** *Outline of the Method*

To a neutralised solution of sodium hydroxide, addition of mercuric thiocyanate and ferric ammonium sulphate, produces a highly coloured ferric thiocyanate complex, the intensity of which is proportional to the chloride content. The absorbance of this colour is measured at 450 nm wave length.

#### **A-4.4.2** *Apparatus*

##### **A-4.4.2.1** *Standard laboratory apparatus*

##### **A-4.4.2.2** *Spectrophotometer or photocolorimeter*

**A-4.4.2.3** *Platinum evaporating dish* — approximately 60 ml capacity. Teflon beaker can also be used.

#### **A-4.4.3** *Reagents*

**A-4.4.3.1** *Ferric ammonium sulphate solution, 0.25 mol/l* - Dissolve 49.02 g of ferric ammonium sulphate dodecahydrate  $[\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$  in 203 ml of dilute nitric acid (about 1.5 mol/l) and dilute to 500 ml with distilled water.

**A-4.4.3.2** *Mercury (II) thiocyanate solution* — saturated solution in absolute alcohol.

**A-4.4.3.3** *Standard chloride solution* — Weigh accurately 0.1649 g of previously dried (at 500°C) and cooled A.R. Sodium Chloride, dissolve in a beaker in distilled water and quantitatively transfer into a one-litre volumetric flask, dilute to mark and mix well. 1 ml of this solution = 100 µg of Cl.

**A-4.4.3.4** *Dilute standard chloride solution* — Transfer 50 ml of standard chloride solution into a 500-ml volumetric flask, dilute with distilled water to the mark and mix well. 1 ml of this solution = 10 µg of Cl.

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**A-4.4.3.5** *Sulphuric acid solution, approximately 1 mol/l* — Pour 56 ml of concentrated sulphuric acid (AR quality) into 944 ml of distilled water slowly, mix and cool.

### **A-4.4.4** *Procedure*

#### **A-4.4.4.1** *Calibration*

Into a series of six 50-ml volumetric flasks, transfer standard chloride solution as given below:

<i>Standard chloride solution, ml</i>	<i>Corresponding mass of chloride (as Cl), mg</i>
0 (compensation)	0
2	20
4	40
6	60
8	80
10	100

Add to each, 2 ml of ferric ammonium sulphate solution and 2 ml of mercury thiocyanate solution in that order. Dilute to mark and mix well. Allow to stand for 15 minutes, transfer to the cell of spectrophotometer and measure absorbance of 450 nm wave length with compensation solution in reference cell.

Plot chloride content in micrograms as abscissa vs. corresponding absorbance values as ordinates.

#### **A-4.4.4.2** *Determination*

Weigh suitable mass of NaOH pellets sample containing 50 to 150  $\mu\text{g}$  of chloride (as Cl) correct to 1 mg and transfer into the platinum evaporating dish, wet this with 15 to 20 ml of distilled water and slowly neutralize with sulphuric acid solution, and add a few drops in excess. Evaporate to nearly dryness on hot water bath. Add 20 to 30 ml of distilled water and quantitatively transfer into a 100-ml volumetric flask, cool and make up to the mark with distilled water and mix well. Filter through Whatman No. 41 or equivalent filter paper and discard a small volume of initial filtrate. Transfer suitable volume (25 to 40 ml) into 50-ml volumetric flask add 2 ml of ferric ammonium sulphate solution and 2 ml

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of mercury thiocyanate solution in that order, dilute to mark with distilled water and mix well. Allow to stand for 15 minutes, transfer to the cell of spectrophotometer. Measure absorbance at 450 nm wave length with distilled water in the reference cell.

### A-4.4.4.3 *Blank test*

Carry out a blank test using exactly the same procedure as used for determination using same quantities of all reagents but omitting the test solution. Instead of test solution use distilled water.

### A-4.4.5 *Calculation*

$$\text{Chloride (as Cl), percent by mass} = \frac{M_1 - M_2}{M_o \times 10\,000}$$

where

$M_1$  = mass in micrograms of chloride corresponding to absorbance of test solution,

$M_2$  = mass in micrograms of chloride corresponding to absorbance of blank solution, and

$M_o$  = mass in grams of sodium hydroxide in the test portion of the aliquot used for colour development.

( CHD 1 )



**AMENDMENT NO. 1 MAY 2003**  
**TO**  
**IS 376 : 1986 SPECIFICATION FOR SODIUM**  
**HYDROXIDE ANALYTICAL REAGENT**  
*( Third Revision )*

( *Pages 6 and 7, clause A-3.4* ) — Substitute the following for the existing clause:

**'A-3.4 Calculation**

Sodium hydroxide (as NaOH), percent by mass	$\frac{[10A + (B - C)] \times N \times 80}{M}$
Sodium carbonate (as Na <sub>2</sub> CO <sub>3</sub> ), percent by mass	$\frac{212 \times (C - B) \times N}{M}$

where

*A* = volume of 1N hydrochloric acid added;

*B* = volume of 0.1 N hydrochloric acid added to complete the phenolphthalein titration;

*C* = volume of 0.1 N hydrochloric acid added to complete the methyl orange titration;

*N* = exact normality of 0.1 N hydrochloric acid; and

*M* = mass, in g, of the sample for preparing the solution of the test.'

( *Page 7, clause A-5* ) — Substitute the following for the existing clause:

**A-5 DETERMINATION OF SULPHATES**

**A-5.1 General**

Two methods have been specified for determination of sulphates by Visual comparison (Method A) and Turbidimetric method (Method B). In case of dispute, Method A shall be the referee method.

**Price Group 3**

## **Amend No. 1 to IS 376 : 1986**

### **A-5.2 Method A (Visual Comparison Method)**

#### **A-5.2.1 Apparatus**

##### **A-5.2.1.1 Nessler cylinders, 50 ml capacity**

#### **A-5.2.2 Reagents**

##### **A-5.2.2.1 Dilute hydrochloric acid, approximately 5N.**

##### **A-5.2.2.2 Barium chloride solution, approximately 10 percent (m/v).**

##### **A-5.2.2.3 Standard sulphate solution**

Dissolve 0.181 4 g of potassium sulphate in water and dilute to 1 000 ml. One millilitre of this solution contains 0.1 mg of sulphate (as  $\text{SO}_4$ ).

#### **A-5.2.3 Procedure**

Transfer 20 ml of the sample solution (A-2.1) to a porcelain dish, add 15 ml of dilute hydrochloric acid and evaporate to dryness on a steam-bath. Dissolve the residue in 15 ml of water and transfer to a Nessler cylinder. Add 2 drops of dilute hydrochloric acid and 2 ml of barium chloride solution. Dilute to 50 ml. Carry out a control test in the other Nessler cylinder using 1 ml of standard sulphate solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the turbidity produced in the two cylinders after 20 min.

**A-5.2.4** The limit prescribed for sulphates shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

### **A-5.3 Method B (Turbidimetric Method)**

#### **A-5.3.1 Outline of the Method**

Neutralization of the test portion (for alkaline salts) with hydrochloric acid and making it acidic with slight excess of hydrochloric acid and precipitation of sulphates as barium sulphate under well defined conditions. Measurement of turbidity using a spectrophotometer (or photocolormeter) at 470 nm. This method is applicable when sulphate content is less than 0.1 percent by mass.

#### **A-5.3.2 Reagents**

##### **A-5.3.2.1 Barium chloride**

A.R. Barium Chloride dihydrate of uniform particle size between 0.5 and 1.25  $\mu\text{m}$ , standardized by screening. It is essential that all preparation

concerning the determination and standardization shall be carried out using a product of the same particle size distribution.

**A-5.3.2.2** *Sodium carbonate solution*, approximately 0.5 mol/l.

**A-5.3.2.3** *Hydrochloric acid solution*, approximately 1 mol/l.

**A-5.3.2.4** *Hydrochloric acid solution*, approximately 6 mol/l.

**A-5.3.2.5** *Standard sulphuric acid solution*

0.05 ml/l. Prepare approximately 1 mol/l solution by pouring 56 ml of concentrated sulphuric acid into 944 ml of distilled water. Dilute 50 ml of this solution to 1 000 ml in volumetric flask which gives approximately 0.05 mol/l solution. Standardize this against standard sodium carbonate solution (0.05 ml/l) prepared by dissolving A.R. Sodium carbonate dried at 260-270°C. Dissolve 1.324 9 g in distilled water, transfer quantitatively into a 250 ml flask, make up to mark and mix well.

**A-5.3.2.5.1** *Standard sulphuric acid solution*, 0.100 g SO<sub>4</sub> per litre.

Transfer 20.8 ml of 0.05 mol/l standard sulphuric acid solution into a one litre volumetric flask, dilute to mark with distilled water and mix well, one millilitre of this solution = 0.100 mg SO<sub>4</sub>.

**A-5.3.3** *Apparatus*

**A-5.3.3.1** *Standard laboratory apparatus*

**A-5.3.3.2** *Platinum evaporating dish*, 60 ml capacity.

**A-5.3.3.3** Spectrophotometer or photometric absorptiometer fitted with filters giving only a negligible transmission below 450 nm and above 550 nm.

**A-5.3.4** *Procedure*

**A-5.3.4.1** *Calibration*

Into each of a series of nine 50 ml one mark volumetric flasks, place the volumes of standard sulphuric acid solution as given below:

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<i>Sulphuric acid solution</i>	<i>Corresponding mass of SO<sub>4</sub></i>
ml	mg
0 (compensation)	0
5.0	0.5
10.0	1.0
15.0	1.5
20.0	2.0
25.0	2.5
30.0	3.0
35.0	3.5
40.0	4.0

To each flask, add 2 ml of sodium carbonate solution and 5 ml of hydrochloric acid solution, stir dilute to flask and mix well.

Transfer 25.0 ml of each of the solution except the first to separate 100 ml dry beaker each containing 0.15 g barium chloride. Stir by hand for 1 min at the rate of 2 revolutions per second. Allow to stand for 15 min at  $27 \pm 2^\circ\text{C}$ .

NOTE — Stagger the test in order to adhere to the contact times indicated.

Stir by hand and transfer a sufficient quantity of each of the solution to a cell of the spectrophotometer and measure the absorbance at 470 nm wavelength. Use compensation solution to adjust optical zero of spectrophotometer.

Plot a graph with SO<sub>4</sub> content, in mg, as abscissae versus corresponding absorbance values as ordinates. It should be noted that the calibration curve is linear only above 0.5 mg of SO<sub>4</sub>.

### A-5.3.4.2 Determination

#### A-5.3.4.2.1 Test portion

Weigh to the nearest 1 mg quantity of the test sample of sodium hydroxide containing 1 to 4 mg of SO<sub>4</sub> and transfer into a platinum evaporating dish. Wet the sample with 10 to 20 ml of distilled water and neutralize with hydrochloric acid solution ( see A-5.3.2.4 ) and add a few drops in excess. Heat the evaporating dish on a water bath to almost dryness and add 5 ml of hydrochloric acid solution ( see A-5.3.2.3 ) and 20 to 30 ml distilled water. Heat again on water bath for a few minutes and transfer quantitatively into a 50 ml volumetric flask, cool, make up to the mark and mix well. Filter through a Whatman No. 41 filter paper, discarding a small volume of the initial filtrate.

#### A-5.3.4.2.2 *Turbidimetric reaction*

Transfer 25 ml filtrate ( *see A-5.3.4.2.1* ) into a 100 ml beaker containing 0.15 g of barium chloride, stir by hand for 1 min at the rate of 2 revolutions per second. The barium chloride should then be in complete solution. Leave undisturbed for 15 min at  $27 \pm 2^\circ\text{C}$ .

#### A-5.3.4.2.3 *Turbidity measurement*

Transfer a sufficient quantity of the test solution to a cell of the spectrophotometer (same size as the one used for calibration) and use to adjust zero of the spectrophotometer.

Stir by hand the turbidity developed solution (*see A-5.3.4.2.2*). Transfer it into a cell (same size as the one used for calibration) and measure absorbance at 470 nm wavelength.

NOTE — If the turbidity developed in the test portion is beyond the limits of the calibration range, dilute an aliquot filtrate (*see A-5.3.4.2.1*) to 50 ml and use 25 ml of this diluted solution to develop turbidity. In this case, use the diluted solution in the reference cell.

#### A-5.3.4.2.4 *Blank test*

At the same time, carry out a blank test following the same procedure and using the same quantities of all reagents used for determination, but by replacing the test portion by 5 ml of standard sulphuric acid solution corresponding to 0.5 mg of  $\text{SO}_4$  to allow operation in the linear part of the calibration curve.

#### A-5.3.5 *Calculation*

The sulphate content is obtained by the following formula:

$$\text{Sulphate (as SO}_4\text{), percent by mass} = \frac{(M_1 - M_2)}{M_0 \times 10}$$

where

$M_1$  = mass of sulphate in mg corresponding to absorbance of the test portion;

$M_2$  = mass of sulphate, in mg, corresponding to the absorbance of the blank solution after deduction of 0.5 mg of  $\text{SO}_4$  added (that is Graph value - 0.5); and

$M_0$  = mass of sodium hydroxide, in g, corresponding to aliquot used for turbidity development.

## **Amend No. 1 to IS 376 : 1986**

( Page 10, clause A-8 ) — Substitute the following for the existing clause:

### **A-8 DETERMINATION OF SILICATES**

#### **A-8.1 General**

Two methods have been specified for determination of silicate by Visual comparison (Method A) and Spectrophotometries method (Method B). In case of dispute, Method A shall be the referee method.

#### **A-8.2 Method A (Visual Comparison Method)**

##### **A-8.2.1 Apparatus**

**A-8.2.1.1 Nessler cylinders** — 100 ml capacity.

##### **A-8.2.2 Reagents**

**A-8.2.2.1 Dilute sulphuric acid** — approximately 5N.

**A-8.2.2.2 Ammonium molybdate solution**

Dissolve 10 g of ammonium molybdate in 100 ml of dilute sulphuric acid (1N).

**A-8.2.2.3 Sodium citrate solution**

Dissolve 8 g of sodium citrate (as  $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ ) in 100 ml of water.

**A-8.2.2.4 Reducing solution**

Dissolve 7 g of anhydrous sodium sulphite in 100 ml of water and add 1.5 g of 1-amino-2-naphthol-4-sulphonic acid. When this is dissolved, add to this solution, a solution containing 90 g of sodium bisulphite in 800 ml of water, and dilute to 1 000 ml.

**A-8.2.2.5 Sodium carbonate, anhydrous.** See IS 296.

**A-8.2.2.6 Standard silicate solution**

Fuse 0.1 g of pure, precipitated silica with anhydrous sodium carbonate in a platinum crucible and leach the melt with water, neutralize with dilute sulphuric acid and dilute to 1 000 ml. Dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution contains 0.01 mg of silica (as  $\text{SiO}_2$ ). Store the solution in a polyethylene bottle.

### **A-8.2.3 Procedure**

Dissolve 10 g of the sample in water in a polyethylene beaker and make up to 250 ml in a standard flask. Transfer 10 ml of the solution into a polyethylene beaker, neutralize with dilute sulphuric acid and add excess of acid to bring the pH of the solution to 1.5 to 1.6. Transfer to a 100-ml Nessler cylinder, add 2 ml of ammonium molybdate solution and allow to stand for 5 min. Then add 5 ml of sodium citrate solution, followed by 2 ml of reducing solution (see A-8.2.2.4). Dilute to 100 ml and stir well. Carry out a control test in another Nessler cylinder, using 4 ml of standard silica solution in place of the sample and the same quantities of other reagents in the same total volume of reaction mixture. Allow the two Nessler cylinders to stand for 30 min and compare the colour developed in the two cylinders.

**A-8.2.3.1** The limit prescribed for silicates shall be taken as not having been exceeded if any blue colour produced in the test with the material is not deeper than that produced in the control test.

## **A-8.3 Method B (Spectrophotometric Method)**

### **A-8.3.1 Outline of the Method**

This method specified a reduced molybdosilicate spectrophotometric method for the determination of soluble silica. This method is applicable for materials with silica content in presence of phosphate.

Depolymerization of any polymerized silica by treatment with hydrofluoric acid and boric acid. Formation of the oxidized molybdosilicate complex (yellow) under well defined conditions of acidity ( $pH\ 1 \pm 0.05$ ).

Selective reduction of the complex in a strong sulphuric acid medium in the presence of oxalic acid to eliminate interference of phosphate.

Spectrophotometric measurement of the coloured complex at a wave length of maximum absorption (about 795 nm).

### **A-8.3.2 Reagents**

#### **A-8.3.2.1 Distilled water**

#### **A-8.3.2.2 Sulphuric acid**

Approximately 2 mol/l. Add 112 ml of concentrated A.R. sulphuric acid (sp gr 1.84) into 888 ml of distilled water slowly by constant stirring, cool and mix well.

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### **A-8.3.2.3** *Sulphuric acid*

Approximately 8 mol/l. Add 448 ml of concentrated A.R. sulphuric acid (sp gr 1.84) to 552 ml of water slowly by constant stirring, cool and mix well.

### **A-8.3.2.4** *Sodium molybdate*

274 g/l solution. Dissolve 27.4 g of sodium molybdate dihydrate ( $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ ) in warm distilled water in a beaker of material free from silica of suitable capacity and after cooling dilute to 100 ml. Store if necessary, in a bottle of material free from silica and filter, if necessary, before use.

### **A-8.3.2.5** *Oxalic acid*

100 g/l solution. Dissolve 10 g of oxalic acid dihydrate ( $\text{COOH} \cdot \text{COOH} \cdot 2\text{H}_2\text{O}$ ) in 100 ml distilled water.

### **A-8.3.2.6** *Ascorbic acid*

25 g/l solution. Dissolve 2.5 g of ascorbic acid in water and dilute to 100 ml. Store the solution in a bottle of material, free from silica and protect from light.

Prepare this solution at the time of use or as an alternative, prepare reduction solution.

### **A-8.3.2.7** *Reduction solution*

**A-8.3.2.7.1** Dissolve 7 g of anhydrous sodium sulphite ( $\text{Na}_2\text{SO}_3$ ) in 50 ml distilled water. Then add 1.5 g of 4-amino-3-hydroxynaphthalene-1-sulphonic acid ( $\text{C}_{10}\text{H}_9\text{NO}_4\text{S}$ ) and dissolve by grinding.

**A-8.3.2.7.2** Dissolve 90 g anhydrous metabisulphite ( $\text{Na}_2\text{S}_2\text{O}_5$ ) in 900 ml of water.

**A-8.3.2.7.3** Mix ( see **A-8.3.2.7.1** and **A-8.3.2.7.2** ) and dilute to 1 000 ml. Filter if turbid and store in a opaque bottle free from silica.

**A-8.3.2.8** *Sodium fluoride solution*, 20 g/l solution.

**A-8.3.2.9** *Boric acid solution*, saturated solution at ambient temperature.

### **A-8.3.2.10** *Standard silica solution*

0.200 g  $\text{SiO}_2$  per litre. Weigh to the nearest 1 mg 0.200 g of  $\text{SiO}_2$  obtained by heating pure silicic acid ( $\text{H}_2\text{SiO}_3$ ) at 1 000°C until constant mass (consecutive weighings should not differ by more than 1 mg) and cool in a desiccator into a platinum crucible, add 2 g of anhydrous  $\text{Na}_2\text{CO}_3$  to the crucible, mix well



preferably with a platinum spatula and carefully fuse the mixture. Add warmwater directly to the crucible, heat gently until the contents are completely dissolved and transfer quantitatively to a beaker of material, free from silica of suitable capacity. Cool, dilute the solution to about 500 ml, transfer quantitatively to a 1 000 ml volumetric flask, dilute to mark and mix well. Transfer the solution immediately to a bottle of material free from silica. one mililitre of this solution is equal to 0.2 mg of  $\text{SiO}_2$ .

**A-8.3.2.10.1** *Standard silica solution*

2.0 mg of  $\text{SiO}_2$  per litre. Dilute 20 ml of standard silica solution (*see A-8.3.2.10*) to 1 000 ml in a volumetric flask. Prepare this solution at the time of use. One mililitre of this solution is equal to 2.0  $\mu\text{g}$  of  $\text{SiO}_2$

**A-8.3.3** *Apparatus*

**A-8.3.3.1** *Standard laboratory apparatus*

**A-8.3.3.2** *pH meter*

**A-8.3.3.3** *Spectrophotometer or photocolorimeter*

**A-8.3.3.4** *Platinum crucible with lid of suitable size*

**A-8.3.4** *Procedure*

**A-8.3.4.1** *Preparation of calibration graph*

**A-8.3.4.1.1** *Preliminary test for control and correction of pH*

Place 10.0 ml of the standard silica solution (*see A-8.3.2.10.1*) in a beaker. Add the distilled water to bring volume to 15 ml and then add 5 ml of sodium molybdate solution, 1 ml of sodium fluoride and 5 ml of boric acid solution. Mix and measure pH with a pH meter. Adjust pH to  $1.0 \pm 0.05$  by adding slowly, using a graduated pipette or a burette drop by drop, mixing after each addition, the necessary quantity of sulphuric acid solution (about 4.5 ml would be required).

Record the actual volume of sulphuric acid required and discard the solution.

**A-8.3.4.1.2** *Preparation of the standard colorimetric solution*

Into a series of six 50-ml beakers, transfer standard silica solution as given below:

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<i>Standard silica solution</i>	<i>Corresponding mass of silica</i>
ml	µg
0 (compensation)	0.0
2.0	4.0
4.0	8.0
6.0	12.0
8.0	16.0
10.0	20.0

Add to each beaker distilled water to make up volume to 15.0 ml. Then add the volume of sulphuric acid solution as calculated above (*see A-8.3.4.1.1*) for pH correction and mix well. Then add 1 ml of sodium fluoride solution, mix and allow to stand for 5 min.

### **A-8.3.4.1.3** *Colour development*

Add to each beaker 5 ml of boric acid solution and 5 ml of sodium molybdate solution mix and allow to stand for 10 min. Add 5 ml of oxalic acid solution and 8 ml of sulphuric acid solution. Allow to stand for 2 minutes. Add 2 ml of ascorbic acid solution or as an alternative 2 ml of reduction solution and allow to stand for 10 min. Transfer the solution quantitatively to a series of 50 ml volumetric flasks, dilute to marks and mix.

### **A-8.3.4.1.4** *Measurement*

Carry out the spectrophotometric measurements with spectrophotometer (or photocolormeter) at 795 nm wavelength, after having adjusted the instrument zero absorbance with the compensation solution, use cells of 4 to 5 cm optical path length.

Plot a graph with µg of SiO<sub>2</sub> contained in 50 ml as abscissae and corresponding absorbance values as ordinates.

### **A-8.3.4.2** *Determination*

#### **A-8.3.4.2.1** *Test solution*

Weigh accurately and correct to 1 mg an amount of sodium hydroxide (containing between 4 to 20 µg of SiO<sub>2</sub>) and transfer into a 100 ml beaker, dissolve in minimum amount of distilled water and neutralize with 1 mol/l sulphuric acid (about 6 ml) and then add 0.5 ml acid excess, heat on a water bath

to expect all CO<sub>2</sub> liberated and cool. Ensure that the total volume is around 15 ml. Then add the volume of sulphuric acid solution ( *see A-8.3.2.2* ) used for pH correction in the preliminary test and 1 ml of sodium fluoride solution, mix and allow to stand for 5 minutes. Develop colour by the procedure given in **A-8.3.4.1.2.**

#### **A-8.3.4.2.2** *Blank test*

Carry out a blank test following the same procedure and using the same quantities of all reagents used for the determination but replacing the test solution by the same volume of distilled water.

#### **A-8.3.4.2.3** *Spectrophotometric measurements*

Carry out the spectrophotometric measurements by the procedure specified in **A-8.3.4.1.4** at 795 nm wave length after adjusting the instrument to zero absorbance against water. Use the same size of the cell used for calibration.

#### **A-8.3.5** *Calculation*

By means of the calibration graph determine the mass of SiO<sub>2</sub> content corresponding to the aliquot used for colour development.

The silica content is expressed by the relation given below:

$$\text{Silica (as SiO}_2\text{), percent by mass} = \frac{(M_1 - M_2)}{M_0 \times 10\,000}$$

where

$M_1$  = mass of  $\mu$  g of SiO<sub>2</sub> in the test portion of the aliquot,

$M_2$  = mass of  $\mu$ g of SiO<sub>2</sub> in the blank; and

$M_0$  = mass of original material in g of the aliquot portion used for colour development.

( *Page 11, clause A-9.2.3* ) — Insert the following caution at the end of the clause:

'*Caution:* Potassium Cyanide is highly poisonous and hence handle with care.'

( *Page 12, clause A-10.1.2.5* ) — Substitute the following for the existing clause:

## **Amend No. 1 to IS 376 : 1986**

### **A-10.1.2.5** *Standard iron solution*

Dissolve 0.702 g of ferrous ammonium sulphate  $[\text{FeSO}_4 (\text{NH}_4)_2 \text{SO}_4]$  in 100 ml of distilled water and 10 ml of concentrated sulphuric acid, add dilute potassium permanganate solution dropwise (0.2 percent, *m/v*) until a slight pink colour persists after stirring, and then dilute with water to 1 000-ml mark. Transfer 100 ml of this solution to 1 000-ml volumetric flask and dilute 100 ml of this solution to 1 000 ml. One millilitre of the diluted solution is equivalent to 0.01 mg of iron (as Fe).'

( CHD 1 )

# *Indian Standard*

## SPECIFICATION FOR

### SODIUM HYDROXIDE, ANALYTICAL REAGENT

### ( *Third Revision* )

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## IS : 376 - 1986

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( Continued on page 21 )

***Indian Standard***  
**SPECIFICATION FOR**  
**SODIUM HYDROXIDE, ANALYTICAL REAGENT**  
**( *Third Revision* )**

**0. FOREWORD**

**0.1** This Indian Standard ( Third Revision ) was adopted by the Indian Standards Institution on 28 February 1986, after the draft finalized by the Acids, Alkalis and Halides Sectional Committee had been approved by the Chemical Division Council.

**0.2** This standard was originally published in 1952 and consequently revised in 1969. It was further revised in 1976. The Committee responsible for the preparation of this standard decided to revise it again on the basis of experience gained by the industry over all these years.

**0.2.1** In the present revision, the limit of calcium has been modified to calcium and magnesium with the corresponding change in the method of test. A new alternate method for estimation of iron has also been prescribed. Sodium hydroxide content has also been revised upwards from 98 to 99 percent.

**0.3** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off values should be the same as that of the specified values in this standard.

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**1. SCOPE**

**1.1** This standard prescribes, the requirements and the methods of sampling and test for sodium hydroxide, analytical reagent.

**2. REQUIREMENTS**

**2.1 Description** — The material shall be in the form of white sticks or pellets.

---

\*Rules for rounding off numerical values ( *revised* ).

## IS : 376 - 1986

**2.2** The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Appendix A. Reference to the relevant clauses of the Appendix A is given in col 4 of Table 1.

**TABLE 1 REQUIREMENTS FOR SODIUM HYDROXIDE,  
ANALYTICAL REAGENT**

SL NO.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST ( REF TO CL No. IN APPENDIX A )
(1)	(2)	(3)	(4)
i)	Sodium hydroxide ( as NaOH ), percent by mass, <i>Min</i>	99.0	A-3
ii)	Carbonate ( as Na <sub>2</sub> CO <sub>3</sub> ), percent by mass, <i>Max</i>	1.0	A-3
iii)	Chlorides ( as Cl ), percent by mass, <i>Max</i>	0.005	A-4
iv)	Sulphates ( as SO <sub>4</sub> ), percent by mass, <i>Max</i>	0.005	A-5
v)	Nitrates ( as NO <sub>3</sub> ), percent by mass, <i>Max</i>	0.000 5	A-6
vi)	Phosphates ( as PO <sub>4</sub> ), percent by mass, <i>Max</i>	0.001	A-7
vii)	Silicates ( as SiO <sub>2</sub> ), percent by mass, <i>Max</i>	0.01	A-8
viii)	Heavy metals ( as Pb ), percent by mass, <i>Max</i>	0.001	A-9
ix)	Iron ( as Fe ), percent by mass, <i>Max</i>	0.001	A-10
x)	Aluminium ( as Al ), percent by mass, <i>Max</i>	0.002	A-11
xi)	Zinc ( as Zn ), percent by mass, <i>Max</i>	0.001	A-12
xii)	Ammonia ( as NH <sub>4</sub> ), percent by mass, <i>Max</i>	0.000 5	A-13
xiii)	Calcium and Magnesium ( as Ca ), percent by mass, <i>Max</i>	0.004	A-14
xiv)	Arsenic ( as As ), percent by mass, <i>Max</i>	0.000 05	A-15
xv)	Insoluble matter, percent by mass, <i>Max</i>	0.01	A-16
xvi)	Nickel ( as Ni ), percent by mass, <i>Max</i>	0.001	A-17
xvii)	Potassium ( as K ), percent by mass, <i>Max</i>	0.02	A-18

NOTE — The results to be reported on as received basis.

## 3. PACKING AND MARKING

**3.1 Packing** — The material shall be packed in air-tight glass or plastics containers which shall be securely closed.



**3.2 Marking** — The containers shall be labelled with the name of the material; manufacturer's name and recognized trade-mark, if any; net mass; and batch number. The maximum limits for the characteristics given in Table 1 and the following caution note shall also be given on the label:

CAUTION — HANDLE WITH RUBBER GLOVES ON, KEEP  
IN TIGHTLY CLOSED CONTAINERS.

**3.2.1** The product may also be marked with Standard Mark.

**3.2.2** The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards.

## 4. SAMPLING

**4.1** Representative samples of the material shall be drawn and their conformity to this specification determined in accordance with the method prescribed in Appendix B.

## APPENDIX A

( *Clause 2.2 and Table 1* )

### METHODS OF TEST FOR SODIUM HYDROXIDE, ANALYTICAL REAGENT

#### A-1. QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water ( *see IS : 1070-1977\** ) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

#### A-2. PREPARATION OF SAMPLE SOLUTION

**A-2.0 General** — Special care shall be taken in sampling to obtain a representative sample and to avoid absorption of water and carbon dioxide by the sample taken.

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\*Specification for water for general laboratory used ( *second revision* ).

**A-2.1 Procedure** — Weigh quickly about 50 g of the material correctly up to second place of decimal in a stoppered weighing bottle and dissolve in cooled water. Cool and dilute the solution to 500 ml in a volumetric flask. Store in a dry bottle fitted with a rubber stopper. Use this sample solution for tests, where specified.

**A-2.1.1** This solution, if not used on the same day, shall be protected from access to carbon dioxide and moisture by fitting the bottle with a suitable absorption tube.

### A-3. DETERMINATION OF SODIUM HYDROXIDE AND SODIUM CARBONATE

#### A-3.1 Reagents

**A-3.1.1** *Standard Hydrochloric Acid* — approximately 1 N.

**A-3.1.2** *Standard Hydrochloric Acid* — approximately 0.1 N. This is prepared by pipetting 25 ml of 1 N hydrochloric acid ( **A-3.1.1** ) and making it up to 250 ml in a volumetric flask.

**A-3.1.3** *Phenolphthalein Indicator* — Dissolve 0.50 g of phenolphthalein in 100 ml of rectified spirit which has been previously neutralized to the indicator.

**A-3.1.4** *Methyl Orange Indicator* — Dissolve 0.1 g of methyl orange in 100 ml of water.

**A-3.2 Preparation of Sample Solution** — Weigh about 25 g of the material correctly up to second place of decimal and dissolve in carbon dioxide free water. Cool to room temperature and make up to 500 ml in a volumetric flask.

**A-3.3 Procedure** — Pipette out 25 ml of the prepared solution ( **A-3.2** ) into a conical flask and dilute with about 50 ml of water. Add 5 drops of phenolphthalein and titrate against 1 N hydrochloric acid up to a little before the end point. Note the volume of acid added ( *A* ). Now complete the titration with 0.1 N hydrochloric acid till the pink colour is discharged. Note the volume of 0.1 N acid added ( *B* ). Then add 2 to 3 drops of methyl orange indicator and continue the titration with 0.1 N hydrochloric acid to an orange red colour. Note the volume of acid required to complete the titration with methyl orange ( *C* ).

#### A-3.4 Calculation

$$\text{Sodium hydroxide ( as NaOH ),} \\ \text{percent by mass} = \frac{(10 A + B - C) \times N \times 80}{M}$$

$$\text{Sodium carbonate ( as Na}_2\text{CO}_3 \text{ ), } \frac{\text{percent by mass}}{\text{percent by mass}} = \frac{212 \times C \times N}{M}$$

where

$A$  = volume of 1 N hydrochloric acid added,

$B$  = volume of 0.1 N hydrochloric acid added to complete the phenolphthalein titration,

$C$  = volume of 0.1 N hydrochloric acid added to complete the methyl orange titration,

$N$  = exact normality of 0.1 N hydrochloric acid, and

$M$  = mass in g of the aliquot taken for the test.

#### A-4. TEST FOR CHLORIDES

##### A-4.1 Apparatus

A-4.1.1 *Nessler Cylinders* — 50 ml capacity.

##### A-4.2 Reagents

A-4.2.1 *Dilute Nitric Acid* — approximately 5 N.

A-4.2.2 *Silver Nitrate Solution* — approximately 4 percent (  $m/v$  ).

A-4.2.3 *Standard Chloride Solution* — Dissolve 0.1649 g of sodium chloride in water and make up the volume to 1 000 ml. Dilute 50 ml of the solution to 100 ml. One millilitre of this solution contains 0.05 mg of chloride ( as Cl ).

**A-4.3 Procedure** — Transfer 10 ml of the sample solution ( A-2.1 ) into a Nessler cylinder and dilute with 20 ml of water. Add 6 ml of dilute nitric acid and 1 ml of silver nitrate solution. Make up the solution to 50 ml with water. Carry out a control test in the other Nessler cylinder using 1 ml of standard chloride solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the opalescence produced in the two cylinders after 5 minutes.

**A-4.4** The limit prescribed for chlorides shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

#### A-5. TEST FOR SULPHATES

##### A-5.1 Apparatus

A-5.1.1 *Nessler Cylinders* — 50 ml capacity.

## A-5.2 Reagents

**A-5.2.1 Dilute Hydrochloric Acid** — approximately 5 N.

**A-5.2.2 Barium Chloride Solution** — approximately 10 percent ( *m/v* ).

**A-5.2.3 Standard Sulphate Solution** — Dissolve 0.1814 g of potassium sulphate in water and dilute to 1 000 ml. . One millilitre of this solution contains 0.1 mg of sulphate ( as  $\text{SO}_4$  ).

**A-5.3 Procedure** — Transfer 20 ml of the sample solution ( **A-2.1** ) to a porcelain dish, add 15 ml of dilute hydrochloric acid and evaporate to dryness on a steam-bath. Dissolve the residue in 15 ml of water and transfer to a Nessler cylinder. Add 2 drops of dilute hydrochloric acid and 2 ml of barium chloride solution. Dilute to 50 ml. Carry out a control test in the other Nessler cylinder using 1 ml of standard sulphate solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the turbidity produced in the two cylinders after 20 minutes.

**A-5.4** The limit prescribed for sulphates shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

## A-6. TEST FOR NITRATES

### A-6.1 Reagents

**A-6.1.1 Dilute Sulphuric Acid** — approximately 5 N.

**A-6.1.2 Concentrated Hydrochloric Acid** — see IS : 265-1976\*.

**A-6.1.3 Standard Potassium Nitrate Solution** — 0.001 N.

**A-6.1.4 Concentrated Sulphuric Acid** — see IS : 266-1977†.

**A-6.1.5 Standard Indigo Carmine Solution** — Dissolve 0.2 mg of indigo carmine in 400 ml of dilute sulphuric acid and add 20 ml of concentrated hydrochloric acid and sufficient dilute sulphuric acid to make up the volume to 1 000 ml. Dilute 100 ml of the solution to 1 000 ml. Standardize the solution so that 100 ml added to 3.3 ml of potassium nitrate solution is just decolourized on adding 13 ml of concentrated sulphuric acid and heating to boiling. One millilitre of this solution is equivalent to 0.002 mg of nitrate ( as  $\text{NO}_3$  ).

**A-6.1.6 Dilute Ammonium Hydroxide Solution** — approximately 5 N.

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\*Specification for hydrochloric acid ( *second revision* ).

†Specification for sulphuric acid ( *second revision* ).

**A-6.2 Procedure** — Transfer 50 ml of the sample solution ( A-2.1 ) into a 250-ml volumetric flask, add 50 ml of dilute ammonium hydroxide solution, make up to the mark and filter by suction. To 20 ml of the filtrate add 5 ml of dilute sulphuric acid, 1 ml of standard indigo carmine solution and 20 ml of concentrated sulphuric acid and heat to boiling.

**A-6.3** The limit prescribed for nitrates shall be taken as not having been exceeded if the blue colour does not entirely disappear.

## **A-7. TEST FOR PHOSPHATES**

### **A-7.1 Apparatus**

**A-7.1.1 Nessler Cylinders** — 50 ml capacity.

### **A-7.2 Reagents**

**A-7.2.1 Dilute Sulphuric Acid** — approximately 5 N.

**A-7.2.2 Phosphate Reagent No. 1** — Dissolve 5 g of ammonium molybdate in 100 ml of 1 N sulphuric acid.

**A-7.2.3 Phosphate Reagent No. 2** — Dissolve 0.2 g of methyl-*p*-aminophenol sulphate ( metol ) and 20 g of potassium metapisulphite in 100 ml of water.

**A-7.2.4 Standard Phosphate Solution** — Dissolve 1.43 g of potassium dihydrogen phosphate in water and dilute to 1 000 ml. Store in a polyethylene bottle. Dilute 10 ml of this solution to 1 000 ml in a measuring flask immediately before use. One millilitre of the diluted solution contains 0.01 mg of phosphate ( as  $\text{PO}_4$  ).

**A-7.3 Preparation of Sample Solution** — Dissolve approximately 2 g of material correctly up to second place of decimal in 20 ml of water in a platinum dish, neutralize with about 10 ml of dilute sulphuric acid; add 2 ml of the acid in excess and dilute to 50 ml. Use this solution for tests in A-7.4 and A-8.3.

**A-7.4 Procedure** — Transfer 25 ml of the sample solution ( A-7.3 ) into a Nessler cylinder. Add 2 ml of dilute sulphuric acid, 1 ml of phosphate reagent No. 1 and 1 ml of phosphate reagent No. 2 and dilute to 50 ml with water. Carry out a control test in the other Nessler cylinder using 1 ml of standard phosphate solution in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Keep both the cylinders in a water-bath at 60°C for 10 minutes and then compare the colour produced in both the cylinders after 5 minutes.

**A-7.5** The limit prescribed for phosphates shall be taken as not having been exceeded if any blue colour developed in the test with the material is not deeper than that produced in the control test.

## A-8. TEST FOR SILICATES

### A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders* — 50 ml capacity.

### A-8.2 Reagents

A-8.2.1 *Dilute Sulphuric Acid* — approximately 1 N.

A-8.2.2 *Ammonium Molybdate Solution* — Dissolve 10 g of ammonium molybdate in 100 ml of dilute sulphuric acid (1 N).

A-8.2.3 *Sodium Citrate Solution* — Dissolve 8 g of sodium citrate ( $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7, 2\text{H}_2\text{O}$ ) in 100 ml of water.

A-8.2.4 *Potassium Metabisulphite Solution* — Dissolve 20 g of potassium metabisulphite and 1 g of hydroquinone in 100 ml of water.

A-8.2.5 *Anhydrous Sodium Carbonate* — See IS : 296-1974\*.

A-8.2.6 *Standard Silicate Solution* — Fuse 0.100 g of pure, precipitated silica with anhydrous sodium carbonate in a platinum crucible and leach the melt with water, neutralize with dilute sulphuric acid and dilute to 1 000 ml. Dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution contains 0.01 mg of silica ( as  $\text{SiO}_2$  ). Store in a polyethylene bottle.

A-8.3 **Procedure** — Transfer 10 ml of the sample solution ( A-7.3 ) into a Nessler cylinder. Add 1 ml of dilute sulphuric acid and 5 ml of ammonium molybdate solution. Allow to stand for 5 minutes and then add 5 ml of sodium citrate solution followed by 5 ml of potassium metabisulphite solution. Dilute to 50 ml. Carry out a control test in the other Nessler cylinder using 4 ml of standard silicate solution in place of the sample and the same quantities of other reagents in the same total volume of the reaction mixture. Keep both the cylinders in a boiling water-bath for 5 minutes, cool and compare the colour produced in the two cylinders after 5 minutes.

A-8.4 The limit prescribed for silicates shall be taken as not having been exceeded if any blue colour produced in the test with the material is not deeper than that produced in the control test.

## A-9. TEST FOR HEAVY METALS

### A-9.1 Apparatus

A-9.1.1 *Nessler Cylinders* — 50 ml capacity.

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\*Specification for sodium carbonate, anhydrous ( *second revision* ).

**A-9.2 Reagents**

**A-9.2.1 Dilute Hydrochloric Acid** — approximately 5 N.

**A-9.2.2 Dilute Ammonium Hydroxide Solution** — approximately 5 N.

**A-9.2.3 Potassium Cyanide Solution** — Dissolve 10 g of potassium cyanide in 100 ml of water.

**A-9.2.4 Acetic Acid** — glacial.

**A-9.2.5 Hydrogen Sulphide Solution** — saturated and freshly prepared.

**A-9.2.6 Standard Lead Solution** — Dissolve 1.60 g of lead nitrate in water and make up the solution to 1 000 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of lead ( as Pb ).

**A-9.3 Procedure** — Transfer 20 ml of the sample solution ( **A-2.1** ) to a Nessler cylinder, add 12 ml of hydrochloric acid, 10 ml of dilute ammonium hydroxide solution and 1 ml of potassium cyanide solution and dilute to 40 ml. Add 5 ml of hydrogen sulphide solution, dilute to the mark and mix well. Carry out a control test using 2 ml of standard lead solution, 1 ml of acetic acid and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in both the cylinders after 5 minutes.

**A-9.4** The limit prescribed for heavy metals shall be taken as not having been exceeded if any colour produced in the test with the material is not deeper than that produced in the control test.

**A-10. TEST FOR IRON**

**A-10.0 General** — Two methods have been prescribed for the estimation of iron. However, in case of dispute, thiocyanate method shall be the referee method.

**A-10.1 Thiocyanate Method**

**A-10.1.1 Apparatus**

**A-10.1.1.1 Nessler cylinders** — 50 ml capacity.

**A-10.1.2 Reagents**

**A-10.1.2.1 Hydrochloric acid** — see IS : 265-1976\*.

**A-10.1.2.2 Ammonium persulphate**

**A-10.1.2.3 Butanolic potassium thiocyanate** — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously till the solution is clear.

---

\*Specification for hydrochloric acid ( *second revision* ).

**A-10.1.2.4** *Dilute sulphuric acid* — approximately 5 N.

**A-10.1.2.5** *Standard iron solution* — Dissolve 0.702 g of ammonium ferrous sulphate in about 100 ml of water containing 10 ml of dilute sulphuric acid ( 4 N ) and dilute to 1 000 ml. Dilute 100 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of iron ( as Fe ).

**A-10.1.3** *Procedure* — Transfer 10 ml of the sample solution ( **A-2.1** ) into a Nessler cylinder and make acidic with hydrochloric acid. Boil for 1 minute and cool to room temperature. Add 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate. Shake vigorously and allow the layers to separate. Carry out a control test using 1 ml of the standard iron solution and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in the butanolic layers after 2 minutes.

**A-10.1.4** The limit prescribed for iron shall be taken as not having been exceeded, if any red colour produced in the test with the material is not deeper than that produced in the control test.

## **A-10.2 Thioglycollic Acid Method**

**A-10.2.1** *Apparatus*

**A-10.2.1.1** *Nessler cylinders* — 50 ml capacity.

**A-10.2.2** *Reagents*

**A-10.2.2.1** *Thioglycollic acid* — containing not less than 97 percent of thioglycollic acid by mass.

**A-10.2.2.2** *Concentrated ammonium hydroxide* — relative density 0.90.

**A-10.2.2.3** *Standard iron solution* — Dissolve 2.81 g of ferrous ammonium sulphate [  $\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$  ] in water, add a few millilitres of dilute sulphuric acid and dilute to 1 000 ml with water.

**A-10.2.2.4** *Dilute standard iron solution* — Dilute 10 ml of the standard iron solution ( **A-10.2.2.3** ) to 1 000 ml with water. The solution should be prepared freshly as required. This solution contains 0.000 004 g of iron per millilitre ( 1 ml = 0.004 mg Fe ).

**A-10.2.3** *Procedure* — Dissolve exactly 2 g of the sample ( as received ) in 10 ml of water and transfer to a Nessler cylinder. Dilute to 20 ml with water and add 4 drops of thioglycollic acid followed by 1 ml of concentrated ammonium hydroxide.

**A-10.2.3.1** Carry out a control test in another Nessler cylinder using 5 ml of dilute standard iron solution and the same quantities of other reagents. Dilute the solution in both the cylinders to 50 ml and mix well.



**A-10.2.3.2** The requirement specified in Table 1 shall be taken as not having been exceeded, if the intensity of colour produced with the material is not greater than that obtained in the control test ( 10 ppm ).

## **A-11. TEST FOR ALUMINIUM**

### **A-11.1 Apparatus**

**A-11.1.1** *Nessler Cylinders* — 50 ml capacity.

### **A-11.2 Reagents**

**A-11.2.1** *Dilute Acetic Acid* — Dissolve 28.5 g of glacial acetic acid in 100 ml of water.

**A-11.2.2** *Ammonium Aurine Tricarboxylate Aluminon Solution* — Dissolve 0.1 g of ammonium aurine tricarboxylate in 100 ml of water.

**A-11.2.3** *Ammonium Carbonate Solution* — approximately 5 N.

**A-11.2.4** *Standard Aluminium Solution* — Dissolve 1.680 g of aluminium ammonium sulphate [  $\text{AlNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  ] and dilute to 1 000 ml. Further dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution contains 0.01 mg of aluminium ( as Al ).

**A-11.3 Procedure** — Transfer 10 ml of the sample solution ( **A-2.1** ) into a Nessler cylinder. Add 10 ml of dilute acetic acid and 1 ml of ammonium aurine tricarboxylate solution. Allow to stand for 5 minutes and then add 10 ml of ammonium carbonate solution. Carry out a control test in the other Nessler cylinder using 2.0 ml of standard aluminium solution, 15 ml of water, 1 g of ammonium acetate, 5 ml of dilute acetic acid and 1 ml of ammonium aurine tricarboxylate solution and adding, after a lapse of 5 minutes, 20 ml of ammonium carbonate solution. Make up the volume in the two cylinders to 50 ml and compare the colour produced in the two cylinders after 5 minutes.

**A-11.4** The limit prescribed for aluminium shall be taken as not having been exceeded, if any pink colour produced in the test with the material is not deeper than that produced in the control test.

## **A-12. TEST FOR ZINC**

### **A-12.1 Apparatus**

**A-12.1.1** *Nessler Cylinders* — 50 ml capacity.

### **A-12.2 Method A**

#### **A-12.2.1 Reagents**

**A-12.2.1.1** *Dilute sulphuric acid* — approximately 5 N, zinc-free.

**A-12.2.1.2 Ammonium thiocyanate solution** — Dissolve 57 g of ammonium thiocyanate ( zinc-free ) in 100 ml of water.

**A-12.2.1.3 *p*-Dimethyl-amine styryl B-naphthaozole methiodide solution** — approximately 0.05 percent ( *m/v* ) in rectified spirit ( *see* IS : 323-1959\* ).

**A-12.2.2 Procedure** — Transfer, by means of a pipette, exactly 20 ml of the sample solution ( **A-2.1** ), into a Nessler cylinder and neutralize with dilute sulphuric acid ( about 10 ml ), adding one drop in excess. Then add one drop of ammonium thiocyanate solution and 0.1 ml of *p*-dimethyl-aminostyryl B-naphthaozole methiodide solution. Carry out a blank in another Nessler cylinder using 30 ml of water, one drop of dilute sulphuric acid, one drop of ammonium thiocyanate solution and 0.1 ml of *p*-dimethyl-aminostyryl B-naphthaozole methiodide solution. Make up the volume in the two cylinders to 50 ml and compare the colour produced immediately.

**A-12.2.3** The limit prescribed for zinc shall be taken as not having been exceeded, if the colour produced immediately in the test with the material is not pink when compared with that produced in the blank test.

**A-12.3 Method B** — This method shall be used when the reagents required for Method A are not available.

#### **A-12.3.1 Reagents**

**A-12.3.1.1 Dilute sulphuric acid** — approximately 5 N.

**A-12.3.1.2 Dilute hydrochloric acid** — approximately 5 N.

**A-12.3.1.3 Potassium ferrocyanide solution** — Dissolve about 4.2 g of potassium ferrocyanide in 100 ml of water.

**A-12.3.2 Procedure** — Transfer, by means of a pipette, 10 ml of the sample solution ( **A-2.1** ), into a Nessler cylinder and neutralize with dilute sulphuric acid ( about 2.5 ml ) adding one drop in excess. Dilute with water to 30 ml. Add 1 ml of dilute hydrochloric acid and 1 ml of potassium ferrocyanide solution. Make up the volume to 50 ml. Carry out a blank in the other Nessler cylinder using the same quantities of reagents in the same total volume of the reaction mixture. Allow both the solutions to stand for 1 hour and compare the opalescence produced.

**A-12.3.3** The limit prescribed for zinc shall be taken as not having been exceed, if the opalescence produced in the test with the material is not greater than that produced in the blank test.

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\*Specification for rectified spirit ( *revised* ).

**A-13. TEST FOR AMMONIA****A-13.1 Apparatus**

**A-13.1.1** *Nessler Cylinders* — 50 ml capacity.

**A-13.2 Reagents**

**A-13.2.1** *Potassium Iodide* — crystals, iodate-free.

**A-13.2.2** *Mercuric Chloride Solution* — saturated.

**A-13.2.3** *Potassium Hydroxide* — solid.

**A-13.2.4** *Nessler's Solution* — Dissolve 10 g of potassium iodide in 10 ml of ammonia-free water, and add to it slowly, with stirring mercuric chloride solution till a slight permanent precipitate forms. Add 30 g of potassium hydroxide, and when it has dissolved, add 1 ml more of the mercuric chloride solution and dilute to 200 ml with ammonia-free water. Allow to settle overnight, decant the clear solution and keep it in a bottle closed with a well-fitting rubber stopper.

**A-13.2.5** *Standard Ammonium Chloride Solution* — Dissolve 0.296 g of ammonium chloride in ammonia-free water and make up the volume to 100 ml. Dilute 10 ml of this solution to 1 000 ml with ammonia-free water. One millilitre of the diluted solution contains 0.01 mg of ammonium ( as  $\text{NH}_4$  ).

**A-13.3 Procedure** — Transfer, by means of a pipette, 20 ml of the sample solution ( **A-2.1** ) into a Nessler cylinder and dilute with 30 ml of water. Then add 2 ml of Nessler's solution. Make up the volume to 50 ml. Carry out a control test in the other Nessler cylinder using 1 ml of standard ammonium chloride solution and 2 ml of Nessler's solution in the same total volume of the reaction mixture. Compare the colour produced in the two cylinders after 5 minutes.

**A-13.4** The limit prescribed for ammonia shall be taken as not having been exceeded, if any colour prescribed in the test with the material is not deeper than that produced in the control test.

**A-14. TEST FOR CALCIUM AND MAGNESIUM****A-14.1 Reagents**

**A-14.1.1** *Dilute Hydrochloric Acid* — 20 percent ( v/v ).

**A-14.1.2** *Concentrated Liquor Ammonia*

**A-14.1.3** *Sodium Sulphide Solution* — 12 percent ( m/v ).

**A-14.1.4** *Standard EDTA Solution* — M/100.

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#### A-14.1.5 Methyl Thymol Blue Indicator

**A-14.2 Procedure** — Dissolve about 10 g of the material weighed correctly upto two places of decimal in 40 ml of water, add 55 ml of dilute hydrochloric acid, cool and add 40 ml of ammonia solution and 5 drops of sodium sulphide solution. Titrate with *M*/100 EDTA, using methyl thymol blue as indicator until the blue solution becomes colourless or grey.

**A-14.2.1** The material shall be taken to have passed the test if not more than 1 ml of standard EDTA is required for the titration.

### A-15. TEST FOR ARSENIC

**A-15.1** Dissolve about 2 g of the material correctly weighed up to two decimal places in 10 ml of water and carry out the test for arsenic as prescribed in IS : 2088-1983\*, using 0.001 32 mg of arsenic trioxide (  $\text{As}_2\text{O}_3$  ) for comparison.

### A-16. DETERMINATION OF MATTER INSOLUBLE IN WATER

#### A-16.1 Reagents

**A-16.1.1 Concentrated Hydrochloric Acid** — see IS : 265-1976†.

**A-16.2 Procedure** — Weigh accurately about 50 g of the material in a 600 ml beaker, add 300 ml of water and stir till dissolved. Add concentrated hydrochloric acid till the solution is just alkaline o phenolphthalein. Bring to boil and allow the solution to settle on the hot-plate for 15 minutes. Filter through a weighed Gooch crucible or tared sintered glass crucible ( G No. 4 ) and wash free from alkali with hot water, allowing the water to drain through completely after each washing. Dry in an oven for 1 hour at 105° to 110°C. Cool in a desiccator and weigh.

#### A-16.3 Calculation

$$\text{Matter insoluble in water, percent by mass} = 100 \times \frac{m}{M}$$

where

*m* = mass in g of the insoluble residue, and

*M* = mass in g of the material taken for the test.

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\*Methods for determination of arsenic ( *second revision* ).

†Specification for hydrochloric acid ( *second revision* ).

## A-17. TEST FOR NICKEL

### A-17.1 Apparatus

A-17.1.1 *Nessler Cylinders* — 50 ml capacity.

### A-17.2 Reagents

A-17.2.1 *Citric Acid*

A-17.2.2 *Dilute Ammonia Solution* — relative density 0.90.

A-17.2.3 *Dimethyl Glyoxime* — 1 percent ( *m/v* ) solution in alcohol.

A-17.2.4 *Standard Nickel Solution* — Dissolve 4.05 g of nickel chloride (  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  ) in 50 ml of hydrochloric acid and dilute with water to make up the volume to 1 litre. Dilute 10 ml of this solution to 1 000 ml. One millilitre of this solution is equivalent to 0.01 mg of nickel ( as Ni ).

**A-17.3 Procedure** — Transfer 10 ml of the sample solution ( A-2.1 ) in a Nessler cylinder and dilute with water to 15 ml. Add 2 g of citric acid and dilute with ammonia solution till the contents become slightly alkaline to litmus paper. Add 1 ml of dimethyl glyoxime and make up the volume to 50 ml with water. Carry out a control test, using 1 ml of standard nickel solution and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced after 5 minutes.

A-17.3.1 The limit prescribed for nickel shall be taken as not having been exceeded, if any pink colour produced in the test with the material is not deeper than that produced in the control test.

## A-18. TEST FOR POTASSIUM

### A-18.1 Apparatus

A-18.1.1 *Flame Photometer* — Equipped with interference filter; atomizer and burner; optical selective device consisting of the reflectors, lenses and diaphragms; and measuring instrument consisting of photocell, amplifier and sensitive galvanometer. The galvanometer scale, which measures the intensity of the radiation transmitted by the element, shall range from 0 to 100.

### A-18.2 Reagent

A-18.2.1 *Standard Potassium Solution* — Weigh exactly 1 g of potassium chloride, dissolve in water and dilute to 1 000 ml in a measuring flask. Further dilute 100 ml of this solution to 1 000 ml. The diluted solution contains 0.01 g of potassium chloride per 100 ml.

**A-18.3 Preparation of Sample Solution** — Dissolve approximately 10 g of the sample, dried and accurately weighed, in minimum quantity of water and dilute to 100 ml in a measuring flask.

**A-18.4 Calibration Graph** — Take 10, 20, 40, 60 and 80 ml of standard potassium solution ( **A-18.2.1** ) and dilute to 100 ml in different measuring flasks. The flasks now contain 0.01, 0.02, 0.04, 0.06 and 0.08 g of corresponding — galvanometer readings as given in **A-18.5** and plot, the concentrations against galvanometer readings in a rectangular co-ordinate graph. Draw a smooth curve over the points which gives a calibration graph in the range 0.01 to 0.1 percent potassium chloride.

### **A-18.5 Procedure**

**A-18.5.1** Insert the potassium filter corresponding to wave-length 767 mm, light the burner fed by illuminating gas ( laboratory gas ), adjust the specified air pressure between 0.5 to 0.6 kg/cm<sup>2</sup>, and maintain the above air pressure constant such that flame is non-luminous by turning the control knob.

**A-18.5.2 Spray** — Water and adjust the pointer to zero in galvanometer scale by zero adjustment knob. Then spray the standard potassium chloride solution and adjust the deflection to maximum ( 100 ) by using sensitivity control knob. Again spray water to see whether the pointer comes to zero, and standard solution to indicate 100. Repeat till water reads zero and standard solution reads 100 with same adjustment during both the operations.

**A-18.5.3** Without altering the earlier adjustment of the instrument, spray various, diluted solutions prepared in **A-18.4** and obtain calibration graph in the range 0.01 to 0.1 percent potassium chloride. After washing with water, spray the sample solution and obtain the galvanometer readings. From the graph, read out the corresponding concentration of potassium chloride in the solution ( *A* ).

### **A-18.6 Calculation**

$$\text{Potassium, percent by mass} = \frac{52.35 A}{M}$$

where,

*A* = concentration of potassium chloride in the sample solution, and

*M* = mass in g of the dried sample taken for the test.

## APPENDIX B

( Clause 4.1 )

### SAMPLING OF SODIUM HYDROXIDE, ANALYTICAL REAGENT

#### B-1. SCALE OF SAMPLING

**B-1.1 Lot** — In any consignment, all the containers of the same size and drawn from the same batch of manufacture shall be grouped together to constitute a lot.

**B-1.2** For ascertaining the conformity of the material to the requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose shall depend on the size of the lot and shall be in accordance with Table 2.

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**TABLE 2    NUMBER OF CONTAINERS TO BE SELECTED**

NUMBER OF CONTAINERS IN THE LOT $N$	NUMBER OF CONTAINERS TO BE SELECTED, $n$
(1)	(2)
Up to 25	2
26 to 50	3
51 to 100	4
101 to 300	5
301 and above	6

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**B-1.3** These containers shall be selected at random from the lot. To ensure randomness of selection, random number table ( see IS : 4905-1968\* ) may be used. In case such a table is not available, the following procedure may be adopted:

Starting from any container in the lot, count them as 1, 2, 3..... up to  $r$  and so on, in one order. Every  $r$ th container thus counted shall be withdrawn to constitute the sample, where  $r$  is the integral part of  $N/n$  (  $N$  being the number of containers in the lot and  $n$  the number of containers to be selected ).

#### B-2. PREPARATION OF COMPOSITE SAMPLES

**B-2.1** Draw with an appropriate sampling instrument small portions of the material from different parts to each selected container. The total quantity of the material to be drawn from each container shall not be less than 150 g.

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\* Methods for random sampling.

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**B-2.2** The material drawn from all the selected containers according to **B-2.1** shall be thoroughly mixed together to constitute a single mass. The total material so obtained shall be divided into three approximately equal parts each of which shall be called a composite sample representing the lot.

**B-2.3** Each of the three composite samples obtained as in **B-2.2** shall be immediately transferred to appropriate sample containers which shall be sealed air-tight immediately after filling and marked with necessary details for identification.

**B-2.4** One of the three composite samples shall be marked for the purchaser, another for the supplier and the third kept as a referee sample.

**B-2.4.1** The referee sample shall be kept at a place and under conditions agreed to between the purchaser and the supplier. The referee sample shall be used in case of dispute.

### **B-3. NUMBER OF TESTS AND CRITERION FOR CONFORMITY**

**B-3.1** Tests for all the requirements of this specification shall be conducted on the composite sample.

**B-3.2** The lot shall be declared as conforming to the requirements of this specification if the composite sample satisfies all the requirements specified in Table 1.



(Continued from page 2)

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